(NASA-CR-150671) SPACE STABLE THERMAL CONTROL COATINGS Triannual Report, 1 Nov. 1977 - 28 Feb. 1978 (IIT Research Inst.) 14 p HC A02/MF A01 CSCL 11D

N78-22160

Unclas
G3/24 15649

IITRI

Contract No. NAS8-31906
Report No. IITRI D6118-24(TAR)

SPACE STABLE THERMAL CONTROL COATINGS

for

National Aeronautics and Space
Administration
George C. Marshall Space Flight Center
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For the Period: 1 November 1977 to 28 February 1978

March, ~~1977~~ 1978

TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

SPACE STABLE THERMAL CONTROL COATINGS

1.0 INTRODUCTION

The potential that zinc orthotitanate possesses as a pigment for spacecraft thermal control applications has been thoroughly demonstrated in past studies at IITRI (Reference 1-3). The practical realization of this potential hinges most importantly on pigment stoichiometry and also on the production process and optimized preparative conditions associated with it. The "MOX" method, i.e., the use of zinc and titanium oxalate precursors, has the distinct advantages of simple and rapid processing, and of controlled pigment particle size.

The primary goals of this program for obtaining a specification quality zinc orthotitanate are:

1) Determination of the chemical identity of TiOX ("titanium oxalate").

2) Effect of Zn/Ti ratio for Zn_2TiO_4 on the reflectance spectra and stability to ultraviolet irradiation in vacuum.

3) Optimized processing parameters for reproducibly obtaining a pigment of the most desirable optical properties and behavior.

The ultimate goal of obtaining a specification thermal control coating appears well within achievable reality. The studies discussed in this report are designed to yield a Zn_2TiO_4 paint as an engineering material for use on future spacecraft.

During this reporting period, study plans were formulated for the final portions of this program. In pursuit of these plans, Zn_2TiO_4 pigment powders have been synthesized and paint studies have been performed. These are described in the following sections.

2.0 PROGRAM PLAN

A steering committee meeting on the subject program was held on December 20, 1977 at the Marshall Space Flight Center. Attendees at this meeting were D.R. Wilkes and D.W. Gates of MSFC, and J.E. Gilligan and Y. Harada of IITRI.

The Proposed Program Plan is shown in Figure 1. It was agreed by the participants in the meeting that this basic outline would be followed. In view of the earlier findings on this program that acid washing of the pigment did not enhance stability to a UV-vacuum environment (see Report No. IITRI D6118-12) such experiments will receive lesser emphasis.

The schedule of studies is to be as follows:

1. Acid wash studies (Dec-Jan)
2. Pigment preparation (Jan)
3. PBR studies (Jan-Feb)
4. Thickness studies (Jan-Feb)
5. Long term test (Feb-Mar. start)
6. Specifications and final report preparation (Mar-June)

The tentative list of samples to be included in the long term test is:

1. Zn/Ti = 1.95, PBR = 7.09
2. Zn/Ti = 2.00, PBR = 7.09
3. Zn/Ti = 1.95, PBR = x (to be determined)
4. Zn/Ti = 2.00, PBR = x (to be determined)
5. Z-93
6. S13G-LO
7. Zn_2TiO_4/602-LO

Others will be added as their potential become evident.

3.0 PAINT STUDIES

The purpose of the paint studies during this period was to examine composition from the standpoint of water content and

FIGURE 1

PROPOSED PROGRAM PLAN

1. Pigment Preparation
    a. $Zn_2(C_2O_4)$ and "TiOX" precipitation
    b. Mixing, Zn/Ti = 1.95 and 2.00
    c. Precalcine and calcine
    d. Evaluation: x-ray analysis, reflectance

2. Paint Studies
    a. Composition
        (1) Maximize water content
        (2) Maximize PBR (pigment to binder ratio)

    b. Application
        (1) Maximize thickness
        (2) Optimize technique

    c. Evaluation
        (1) Physical integrity
        (2) Reflectance

3. Paints For Long Term Tests
    a. Calcination: 900°C/8 hr.
    b. Zn/Ti Ratio: 1.95 and 2.00
    c. PBR: 7.09 and x (to be determined in 2.a (2) above)

PBR. Paint formulations were prepared by ball-milling of pigment-PS7 potassium silicate-distilled water mixtures of varying PBR for 6 hours. Coatings were applied by conventional spray painting techniques and cured in air at room temperature. The effect of the compositional variables on paint sprayability and on resultant coatings was determined.

3.1 Water Content Studies

Previous work with Zn_2TiO_4-silicate paints had shown a very "wet" application during spraying, indicating a need for decreased water content. Based on 100 grams of pigment, the PS7 + water amounted to 96 cc H_2O for the original compositions. Reduction of this amount to 76 cc H_2O yielded a formulation of higher viscosity with better spraying characteristics.

In the current studies, even greater reduction in water content was investigated. Compositions which were examined are listed in Table I. In the spraying of these paints, compositions "A" and "B" showed good spraying characteristics while "C" went on very dry. The glossy appearance, which signals the end of a spray cycle with silicate paints, could only be achieved for "C" by a combination of opening of the spray nozzle with contraction of the gun-to-substrate distance by about 50%.

Subsequent examination of cured coatings showed Series "A" and "B" to be uniform and smooth in appearance. In contrast, some of the "C" samples exhibited a very rough appearance which often results in "dry" spraying. Weight and thickness measurements showed that a heavier coating was achieved with fewer spray cycles for "C". A plot of coating thickness vs. weight (Figure 2) for various paints showed that "C" samples with the most uneven surface texture tended to be thicker for the same weight as compared to the other samples.

From these results, it would appear that a water content of about 66 cc per 100 gms pigment is ideal for good sprayability along with achieving a good coating. This contrasts with Z93

TABLE I

PAINT COMPOSITIONS* FOR WATER CONTENT STUDIES

COMPONENT	A	B	C
Zn_2TiO_4	20 gms	20 gms	20 gms
PS7	6 cc	6 cc	6 cc
H_2O	10 cc	8 cc	6 cc
Water Content**	76	66	56

*PBR = 7.09, Zn/Ti ratio = 1.95

*Based on 100 gms pigment

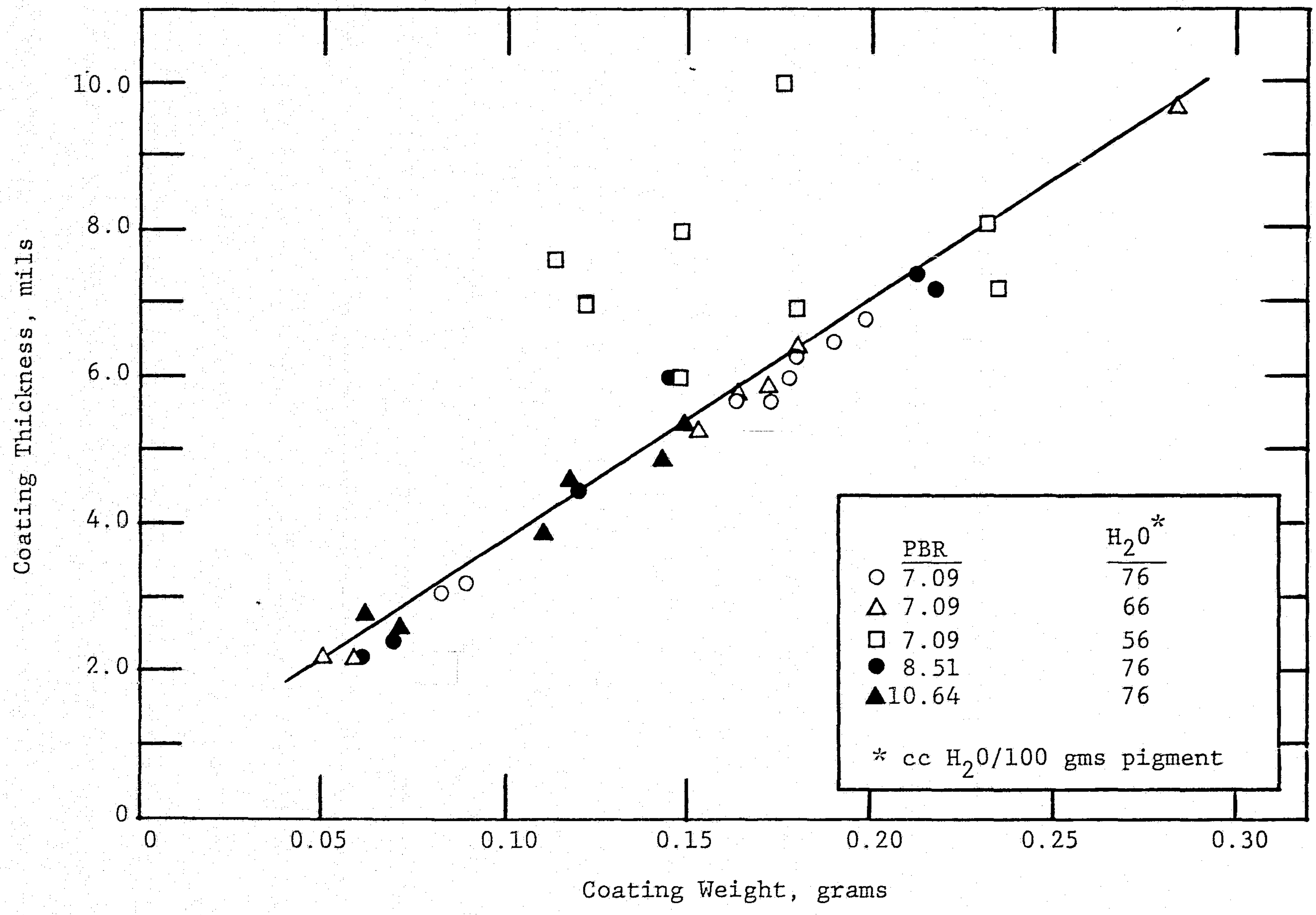

FIGURE 2. COATING THICKNESS VS. WEIGHT FOR ZINC ORTHOTITANATE-SILICATE PAINTS.

(silicate-bonded ZnO) for which 96 cc of total water is required for 100 gms pigment, and can be attributed to the coarser particle size of Zn_2TiO_4 requiring lesser amounts of liquid for good wetting.

3.2 PBR Studies

The PBR experiments consisted of increasing the pigment loadings beyond the previous high of 7.09, to 8.51 and 10.64. Sprayability of these pigment-rich formulations was quite good. Examination of the cured coatings showed them to be somewhat softer than the 7.09 PBR samples. However, the integrity of these coatings appeared to be adequate; good bonding to the aluminum substrate was achieved.

Samples from these PBR studies along with those from the water content studies are now being measured for reflectance.

3.3 UV-Vacuum Studies

The results of a 1000 ESH test for various silicate-bonded Zn_2TiO_4 coatings are shown in Table II. These data were generated by MSFC in the latter part of 1977 and are being presented in this report for documentation. The following sample variables were examined in this test:

1. Pigment-to-binder ratios of 4.26, 5.32, and 7.09.
2. Pigment calcination temperatures of 900° and 1050°C.
3. Zn/Ti ratios for the pigment of 1.95 and 2.00.

As the data show, all samples exhibited excellent stability. Changes in α were all less than 0.01. In view of these very limited $\Delta\alpha$'s, behavioral trends as a function of the above listed variables are not evident.

TABLE II

STABILITY OF Zn_2TiO_4-SILICATE PAINTS

SAMPLE NO.	Zn/Ti RATIO	CALC. TEMP, °C	PBR	$\alpha_{INITIAL}$	$\Delta\alpha_{1000\ ESH}$
M2	1.95	900	4.26	.192	.006
M6	1.95	900	5.32	.169	.006
M10	1.95	900	7.09	.153	.004
M4	1.95	1050	4.26	.228	.004
M8	1.95	1050	5.32	.203	.003
M12	1.95	1050	7.09	.205	.005
M17	2.00	900	4.26	.190	.006
M14	2.00	900	5.32	.183	.003
M22	2.00	900	7.09	.154	.003
M20	2.00	1050	4.26	.230	.008
M16	2.00	1050	5.32	.225	.004
M23	2.00	1050	7.09	.198	.006

3.4 Potential Hardware Applications

Interest in silicate-bonded Zn_2TiO_4 paints appears to be growing in sectors outside of NASA. As reported in IITRI D6118-17 (MLR), samples were submitted to Mr. Carl Maag of Aerojet Electrosystems Company. Aerojet tests revealed goodmechanical properties for this paint.

More recent communication has been with M. Schellhase of Sandia Laboratories, G. Borson and R. Champetier of Aerospace Corporation, and A. Rubin of Aerojet, as well as with Mr. Maag. Paint samples were prepared on Kevlar composite substrates supplied by Sandia. Good bonding was achieved on this polymeric material which was abraded with # 60 cloth, washed with an alkaline detergent, dried, and primed with PS7 prior to paint application.

There is a good possibility that IITRI's Zn_2TiO_4-silicate coating may be specified for flight hardware with which the aforementioned people are involved. It is recognized by the parties involved that the paint is still developmental and has not reached an engineering specification stage.

4.0 SUMMARY

We are now in the final portions of this program. The last major item will be a long term UV-vacuum test involving the best paint candidates in terms of ease of applicability, mechanical properties, and high reflectance. These samples are now being prepared.

Respectfully submitted,
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